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THE INTRODUCTION OF DISLOCATIONS AND SLIP BANDS IN MOLYBDENUM SINGLE CRYSTALS*

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ABSTRACT

The characteristics of selectively introduced slip bands and individual dislocations were investigated. It was found that slip bands produced by indenting the (112) surface had three types of Burgers' vectors concentrated at the two edges of the slip band while the middle of the slip band consisted of primarily the $\pm\frac{1}{2}[11\bar{1}]$ Burger's vector. Scratching the specimen with a needle almost parallel to the (112) surface in the $[11\bar{1}]$ direction produced individual screw dislocations near the scratch mark. Similar scratches normal to the $[11\bar{1}]$ direction produced fine slip bands with a $\pm\frac{1}{2}[11\bar{1}]$ Burger's vector.

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I. INTRODUCTION

A method of introducing fresh dislocations or groups of dislocations (slip bands) of the proper orientations into crystals is essential in dislocation mobility investigations. In general, such techniques as scratching, indenting or ball rolling on the surface of the crystals are used to introduce dislocations selectively.[1-11] For b.c.c. crystals, scratches are usually made on an observation surface (112) or nearly (112) oriented, and in a direction making a 45° angle with the $[\bar{1}\bar{1}1]$ direction (see Figure 1). Indentation with a sharp tool on similar surfaces are also commonly used techniques. Dislocation half loops introduced by these techniques are thought to have a $\pm \frac{1}{2}[11\bar{1}]$ Burger's vector and lie on the $(1\bar{1}0)$ plane[2, 7, 12, 14] and/or on planes of the $\{112\}$ type.[6] Dislocations emerging from the observation surface as revealed by etch-pitting are predominately edge dislocations. Therefore mobility data on these dislocations are limited to edge dislocation investigations. Since the mobility and the asymmetric effects of the screw dislocations are of considerable interest, it is desirable to explore methods to introduce screw dislocations, so that the intriguing characteristics of the screw component can be studied.

For molybdenum single crystals, there have been conflicting reports on the effectiveness of introducing fresh dislocations by scratching the surface with a sharp object.[6,7] Since the Berg-Barrett X-ray topographic technique has demonstrated its accuracy in measuring slip bands in molybdenum crystals,[13] it is the objective of this study to investigate the effectiveness of various techniques to introduce individual dislocations and slip bands and the nature of their Burgers' vectors, using the X-ray topographic method.

II. EXPERIMENTAL PROCEDURE

A (112) surface of commercially available molybdenum single crystals was prepared by wire cutting (South Bay Technology Model 850 Slicing and Dicing Instrument) followed by electrolapping with a NaSCN solution (South Bay Technology Model 451 Facing Instrument). Fresh dislocations or slip bands were introduced into the crystal by (1) indenting the surface with a miniload hardness tester (Leitz Wetzlar). The tip of the diamond indenter has a pyramidal shape. The loading mode is static and the load for indentation can be varied from 5 gm to 500 gm. (2) scratching the surface with the diamond indenter perpendicular to the surface (Figure 2-a),(3) scratching the surface with a needle almost parallel to the surface (Figure 2-b). The Berg-Barrett X-ray (CuKα radiation) exposures were taken with different diffracting conditions (different $\vec{g}$ vectors) to determine the Burgers' vectors. Because of the small subgrains in the crystal, sometimes it was necessary to rock the crystal during exposure so that the entire crystal diffracted. The crystal surface was then electrolapped in small increments to investigate the penetration depths of the fresh dislocations of slip bands.

III. EXPERIMENTAL RESULTS AND DISCUSSION

Experimental results[5,6] reported in the literature assume that slip bands introduced in niobium and molybdenum single crystals by indenting the (112) surface have a single $\frac{1}{2}[11\bar{1}]$ Burger's vector, and that they lie on the $(1\bar{1}0)$ slip plane. X-ray topographic investigations revealed a more complicated situation. When an indentation was made on the (112) surface, all four Burgers' vectors were activated. Extinction experiments indicated that dislocations with three types of Burgers' vectors ($\frac{1}{2}[111]$, $\frac{1}{2}[\bar{1}11]$, and $\frac{1}{2}[1\bar{1}1]$) were concentrated on the two edges of the slip band while the middle of the slip band consisted primarily of dislocations with the $\frac{1}{2}[11\bar{1}]$ Burger's vector. An example is shown in figures 3-a and 3-b. The diffraction vector for figure 3-a was [321] which extincts the $\frac{1}{2}[\bar{1}11]$ Burger's vector, the slip bands were shown as dark contrast. The diffraction vector for figure 3-b is [202] which extincts the $\frac{1}{2}[11\bar{1}]$ Burger's vector, the dark contrast in the middle of the slip bands disappeared, indicating the dislocations in the middle of the slip band were of the $\frac{1}{2}[11\bar{1}]$ Burger's vector. The dark contrast on the edges of the slip bands could not be extincted by any single diffracting condition, suggesting the other three types of Burgers' vectors were present simultaneously. Relatively the same structure of slip bands was formed irrespective of the shape of the indenter (needle or diamond pyramid) or the mode of loading (static or dynamic).

The slip planes on which these slip bands lay could not be identified inequivocally because there was only one slip trace on one observation surface. Examination of the (112) stereographic projection suggests that there are at least 11 possible slip planes of the $\{110\}$, $\{112\}$, or the $\{123\}$ type making an identical slip trace on the (112) observation surface. In addition the slip bands may even lie on the (112) observation plane, although it is unlikely. The assumed $(1\overline{1}0)$ slip plane[6,7] (maximum resolved shear stress plane) reported in the literature for three or four point bending tests, is, therefore, in doubt, since it is well known that the schmid law does not always hold for b.c.c. crystals.[15]

The width of the slip bands (width of the dark contrast) is usually of the same size as the indentation mark, commonly of the order of 30 μm for an indentation made by the miniload hardness tester with a 100 gm load. The length and the penetrating depth of the slip band also depend on the loading conditions, typically of the order of several hundred microns and 60 to 80 μm respectively for an indentation of a 100 gm load.

Scratching the surface with the diamond indenter or with a sharp needle normal to the surface did not produce any well-defined slip bands extending from the scratch. Although there appeared to be some dislocation contrast very near to the scratch mark.

Scratching the (112) surface with a needle almost parallel to the surface produced some very interesting phenomena. When such a scratch was made in a direction normal to the $[11\overline{1}]$ direction (direction of the Burger's vector lying on the (112) plane), fine and sharp slip bands were emitted from the scratch mark (see Figure 4). Extinction experiments showed that these slip bands were of the $\pm \frac{1}{2}[11\overline{1}]$ Burger's vector. The length of these slip bands was approximately 100 μm and and the width 10 μm, and they lay within 4 to 6 μm from the free surface. It is not certain whether these slip bands were groups of dislocation loops lying on the (112) slip plane or they lay on a slip plane making an angle with the (112) observation surface. If the slip bands were indeed surface dislocation loops lying on the (112) plane, then the length to width ratio of these bands implies that the mobility of edge dislocations is approximately ten times faster than that of the screw dislocations at room temperature. It is interesting to note that scratching the specimen with a tool perpendicular to the surface did not produce any well-defined slip bands.

Scratching the specimen with a needle almost parallel to the surface and in the $[11\bar{1}]$ direction produced straight individual dislocations parallel to and extending beyond the scratch mark. In addition, long and straight slip bands extending from the tip of the scratch were also observed (see Figure 5-a). The width of the dislocation contrast was approximately 1.5 to 3 μm and the outermost dislocation was situated approximately 40 μm from the edge of the scratch. These dislocations disappeared after removal of approximately 4 to 6 μm from the specimen surface. Extinction experiments indicated that the Burger's vector for the individual dislocations and for the slip band extending from the scratch tip was the $\frac{1}{2}[11\bar{1}]$ vector (see Figure 5-b). These observations suggest that the individual dislocations along the direction of the scratch (the $[11\bar{1}]$ direction) are screw dislocations and that they are surface dislocations lying on the (112) plane (parallel to the observation surface). If the fine dislocation contrast was due to slip bands lying on a tilted slip plane with respect to the (112) plane, we would expect the depth of these slip bands to be at least of the same order as the distance from the outermost dislocation contrast to the edge of the scratch mark (40 μm). Since the contrast disappeared when a layer of material 4 to 6 μm thick was removed, it is most likely that the contrasts observed on the X-ray topograph are due to single individual screw dislocations lying very close to the surface and on the (112) plane. Screw dislocations introduced by this technique will facilitate the investigation of screw dislocation mobility.

SUMMARY

Indentation of the (112) surface of molybdenum single crystals with a sharp tool produces slip bands extending in the $[11\overline{1}]$ direction. All four types of Burgers' vectors are activated. Dislocations with three types of Burgers' vectors ($\frac{1}{2}[111]$, $\frac{1}{2}[\overline{1}11]$, $\frac{1}{2}[1\overline{1}1]$) are concentrated on the two edges of the slip band while the middle of the slip band consists primarily of dislocations of the $\frac{1}{2}[11\overline{1}]$ Burger's vector. Scratching the specimen with a sharp tool normal to the (112) surface does not produce well-defined slip bands. Scratching the specimen with a needle almost parallel to the (112) surface and in a direction normal to the $[11\overline{1}]$ direction produces fine slip bands with a $\pm\frac{1}{2}[11\overline{1}]$ Burger's vector. The length to width ratio of the slip bands seems to suggest the edge mobility is about ten times as that for screw dislocations at room temperature. Scratching the specimen with a needle almost parallel to the (112) surface and in the $[11\overline{1}]$ direction, produces long individual screw dislocations near the scratch mark. Fine slip bands extending from the tip of the scratch mark are also observed. Experimental observations strongly suggest that these dislocations are located very near the free surface and lie on the (112) plane.

REFERENCES

1. W. G. Johnston and J. J. Gilman, J. Appl. Phys. 30, 129 (1959).
2. D. F. Stein and J. R. Low, Jr., J. Appl. Phys. 31, 362 (1960).
3. J. S. Erickson, J. Appl. Phys. 33, 2459 (1962).
4. H. W. Schadler, Acta Met. 12, 861 (1964).
5. H. D. Guberman, Acta Met. 16, 713 (1968).
6. H. L. Prekel, A. Lawley and H. Conrad, Acta Met. 16, 1089 (1964).
7. G. C. Das and P. L. Pratt, Proceedings of the Second International Conference on the Strength of Metals and Alloys, ASM 1, 103 (1970).
8. A. P. L. Turner and T. Vreeland, Jr., Acta Met. 18, 1225 (1970).
9. T. Vreeland, Jr., Chapter 12 in Techniques of Metal Research, Volume II, Part 1, edited by R. F. Bunshah, Interscience Publishers (1968).
10. D. P. Pope and T. Vreeland, Jr., Phil. Mag. 20, 1163 (1969).
11. K. M. Jassby and T. Vreeland, Jr., Phil. Mag. 21, 1147 (1970).
12. G. C. Das and P. L. Pratt, Mat. Sci. Bull. 2, 487 (1970).
13. R. F. Boyce and T. Vreeland, Jr., Mat. Sci. Eng. 9, 56 (1972).
14. H. L. Prekel and A. Lawley, Phil. Mag. 14, 545 (1966).
15. H. W. Christian, Proceedings of the Second International Conference on Strength of Metals and Alloys, ASM 1, 29 (1970).

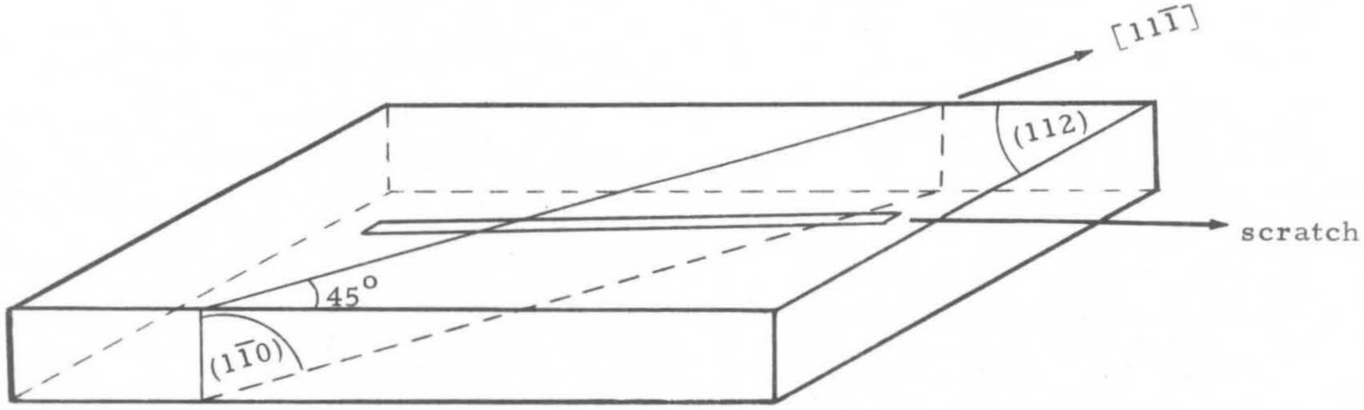
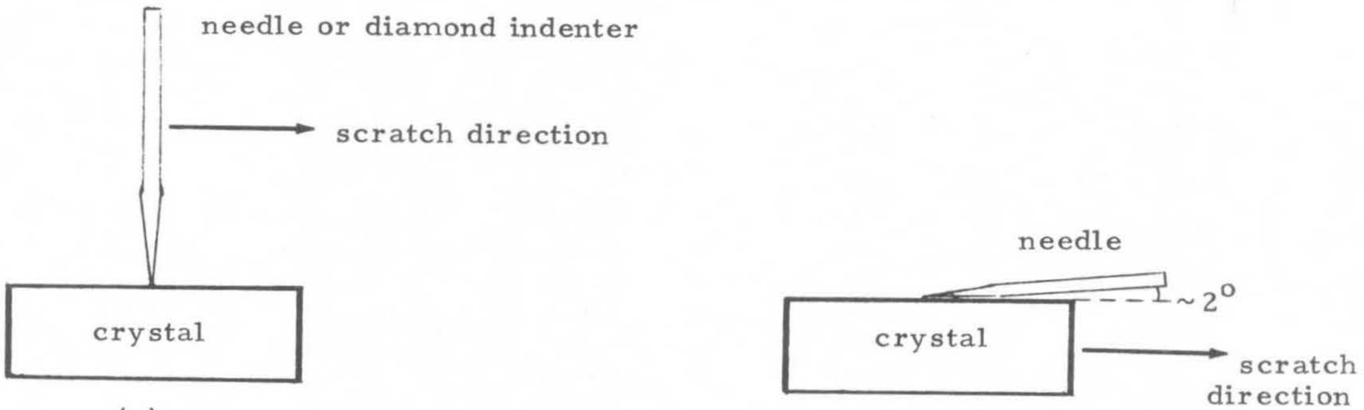


Figure 1



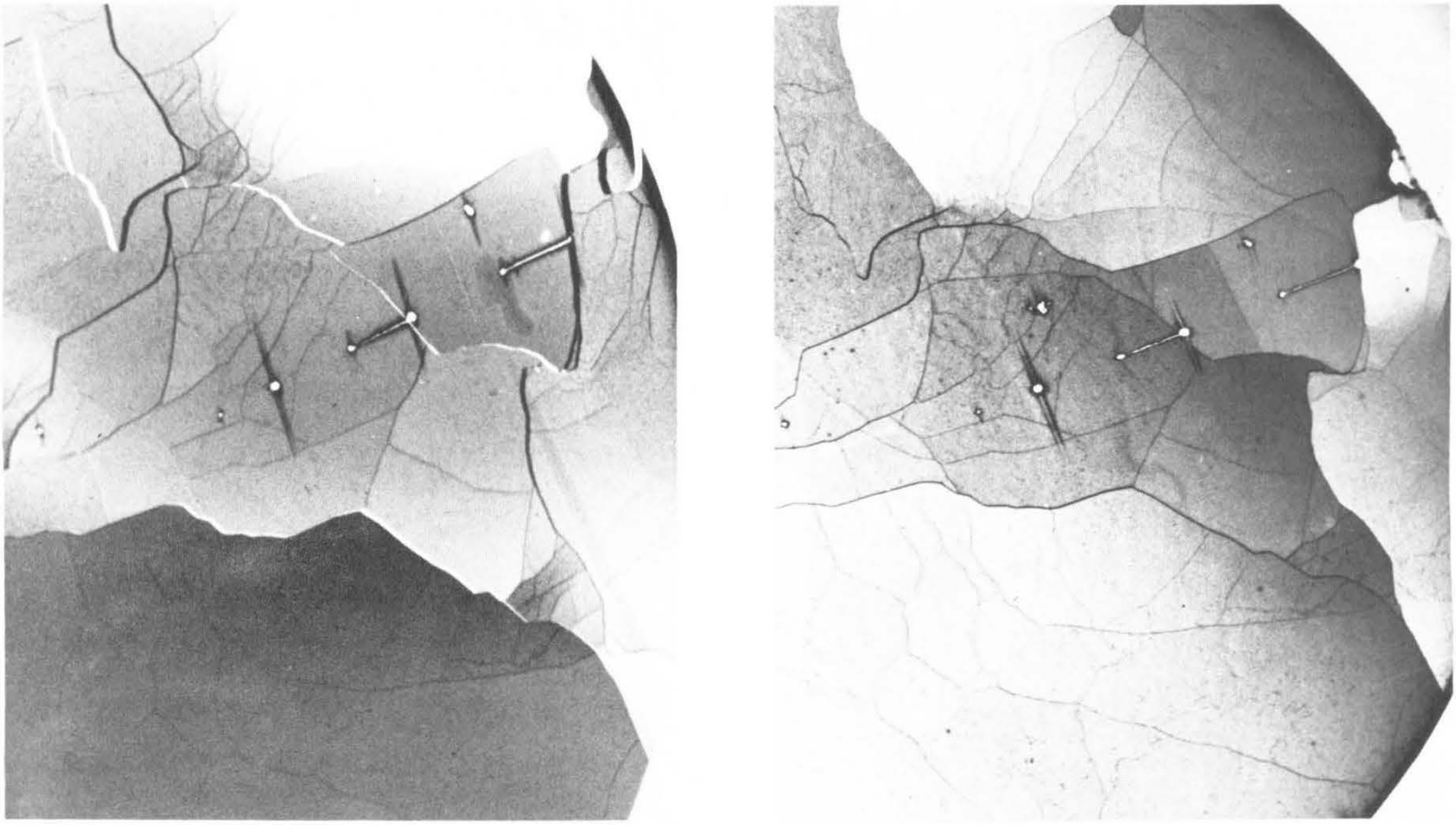
(a)

needle normal to surface

(b)

needle parallel to surface

Figure 2



(a)

$\vec{g} = [321]$

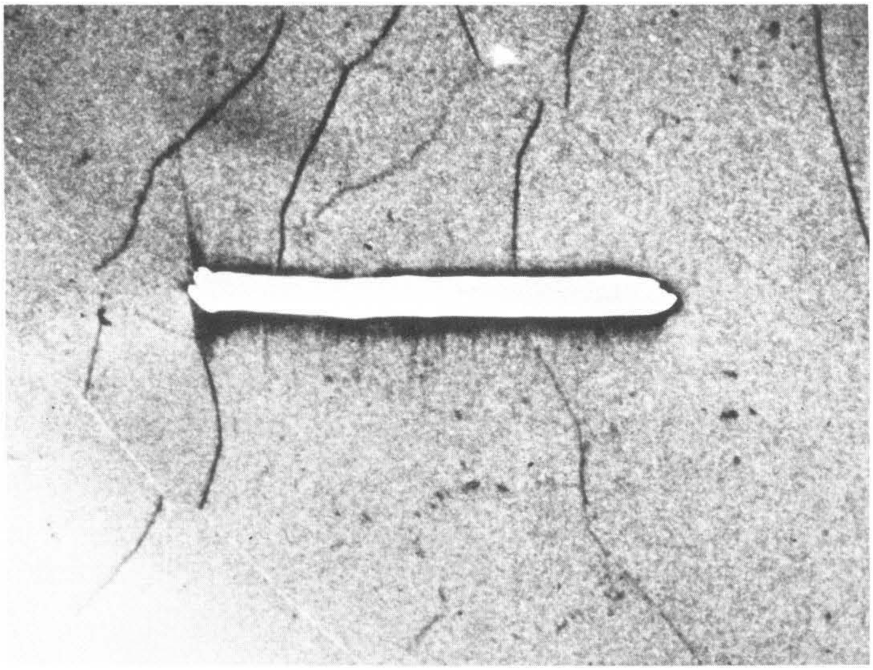
Berg-Barrett topograph (~ 100 X) showing slip bands introduced by indentation.

(b)

$\vec{g} = [202]$

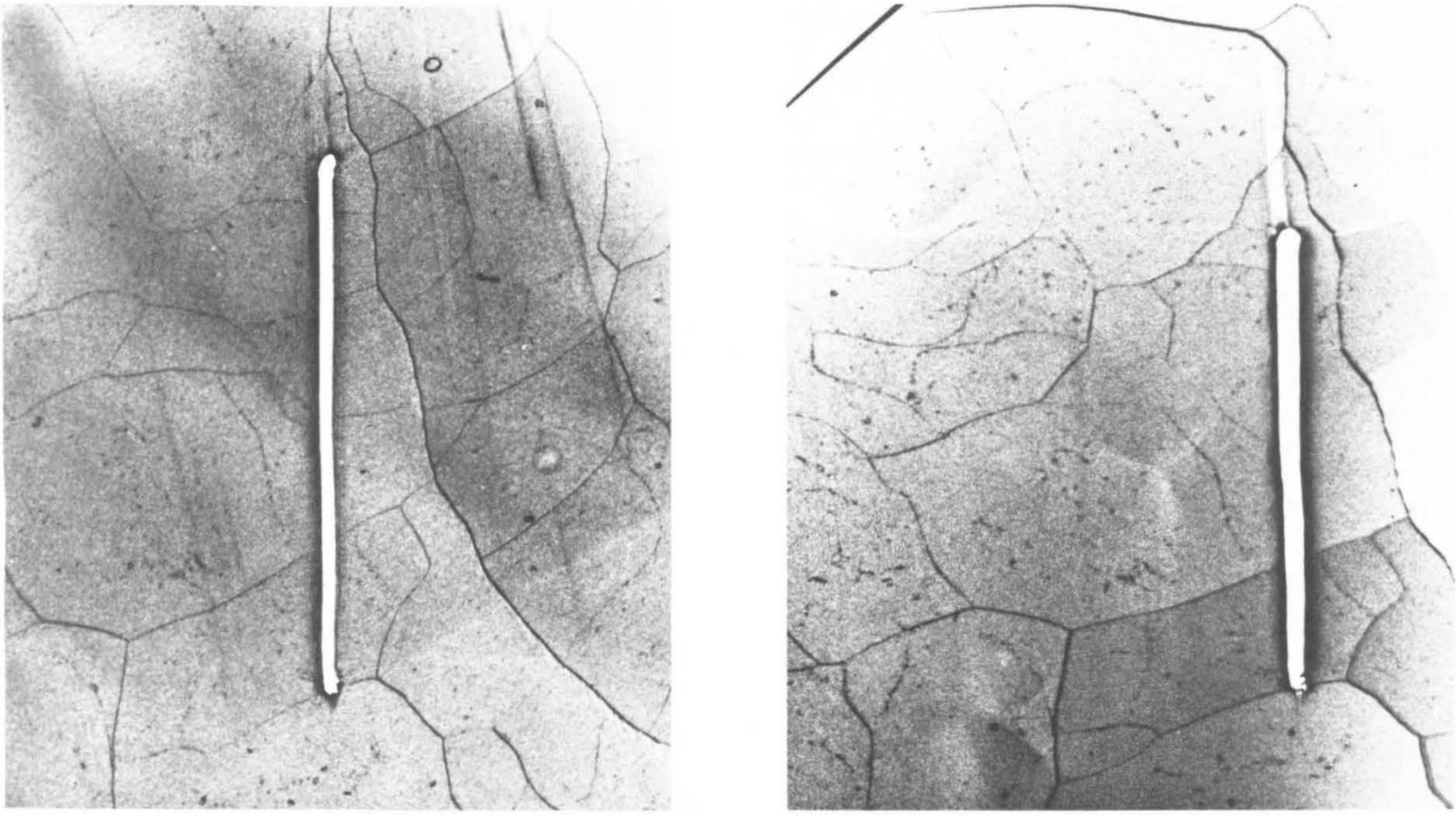
The dark contrast in the middle of the slip bands (shown in a) was extincted by $\vec{g} = [202]$, indicating the dislocations were of the $\pm \frac{1}{2} [11\bar{1}]$ Burger's vector.

Figure 3



Berg-Barrett topography (~ 65 X) of fine slip bands produced by scratching the (112) surface with a needle almost parallel to the surface and in a direction normal to the $[11\bar{1}]$ direction. $\vec{g} = [\bar{2}13]$

Figure 4



$[11\bar{1}]$

(a)

$\vec{g} = [\bar{2}13]$

Berg-Barrett topograph (~ 30 X) of long individual dislocations and slip bands produced by scratching with a needle almost parallel to the (112) surface and in the $[11\bar{1}]$ direction.

$\vec{b}$

$\vec{g}$

(b)

$\vec{g} = [3\bar{1}2]$

The dislocation contrast was extincted by $\vec{g} = [3\bar{1}2]$, indicating the dislocations are screw dislocations with a $\frac{1}{2}$ $[11\bar{1}]$ Burger's vector.

Figure 5